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General rules for mass spectrometric analysis

质谱分析方法通则

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General rules for mass spectrometric analysis

1 Scope

This Standard specifies the general methods of using mass spectrometers to perform mass qualitative analysis and quantitative analysis.

This Standard applies to qualitative analysis and quantitative analysis of mass spectrometry.

2 Terms and definitions

The following terms and definitions are applicable to this document.

2.1 Background

Under the same conditions as the analyzed sample, the mass spectrum signal that is generated when the sample is not fed.

2.2 Interference

Factors that affect the accuracy of analysis results when analyzing a component in a mixed sample.

2.3 Mass-to-charge ratio

The ratio of the mass of ion (measured in relative atomic mass units) to the charge it carries (measured in electron charge); it is abbreviated m/z .

2.4 Mass range

A range between the lower and upper limits of the mass-to-charge ratio that the mass spectrometer can measure.

2.5 Sensitivity

Under the specified conditions, for a certain mass spectrum peak that is generated by the selected compound, the response value of the instrument to the unit sample.

2.6 Resolution

Under the given sample conditions, the separating capacity of the instrument to two adjacent mass spectrum peaks. For two adjacent mass spectrum peaks of equal height, when the peak valley is not greater than 10% of the peak height,

The sampler is a device that introduces the sample into the ion source of the instrument. The sampler is required to introduce the sample into the ion source without changing the structure and composition of the sample and under the vacuum state of the mass spectrometer. Commonly used samplers include diffusion sampling system, direct sampling system and chromatographic sampling system.

4.2.2 Diffusion sampling system

It is generally composed of sample introduction device, gas storage tank, sample measurement device, evacuation and heating device, and corresponding control valves. It is required that the pressure of the storage tank does not change significantly during the measurement process, so as to obtain a stable ion flow. This system is mostly suitable for gas, low-boiling liquid or medium-vapor pressure solid samples without further separation.

4.2.3 Direct sampling system

Commonly used direct sampling method includes probe sampling and injection sampling.

The probe sampling system is composed of probe rod, heating wire, sample cup and vacuum locking system. Place the sample in a small cup at the top of the probe rod; after directly placing the probe into the ion source through the sample inlet of the mass spectrometer, heat the ion source until the sample volatilizes. It is mostly used for liquid or solid pure compounds that are hard to volatile.

For injection sampling, generally introduce the sample solution directly into the ion source through a syringe or an injection pump in a manual or automatic manner. It is mostly used for mass spectrometer tuning and solution sample analysis.

4.2.4 Chromatographic sampling system

For mixture samples of more complex components, it is necessary to use a chromatograph to separate the sample into single components before it enters the ion source of the mass spectrometer. For gas chromatography-mass spectrometry instrument, it generally introduces the analyte from the outlet of the chromatographic column to the ion source through interfaces such as direct introduction, open split flow, or jet separator; for liquid chromatography-mass spectrometry instrument, it mainly uses spraying techniques such as electrospray, thermo-spray and ion-spray to realize sample injection.

4.3 Ion source

4.3.1 Overview

Atmospheric pressure chemical ionization is commonly used in liquid chromatography-mass spectrometry instruments. The corona needle discharges to ionize the solvent; the charged solvent molecules react with the sample molecules to ionize the molecules, which transfers the charge to the sample molecules, so as to ionize the sample molecules. Atmospheric pressure chemical ionization is also a "soft ionization" method, which is suitable for the analysis of non-polar and moderately polar organic compounds.

4.3.6 Matrix assisted laser desorption ionization (MALDI)

It is the cocrystallization film that is formed when using laser to irradiate the sample and the matrix. The matrix absorbs energy from the laser and transfers it to the sample molecules to initiate ionization and desorption of the two; it also has a charge transfer reaction, which ionizes the sample molecules. This ionization source can obtain a large number of single-charged ions, which is a soft ionization technology. The ionization source is widely used in the analysis of large biological molecules and synthetic polymers.

4.3.7 Inductively coupled plasma (ICP)

The inductively coupled plasma is composed of radio frequency generator, work coil and working gas. The radio frequency generator passes the high-frequency current through the work coil to generate a strongly oscillating ring magnetic field in the direction of the axis of the coil; when argon is introduced into the torch tube, the high-frequency spark discharge of the igniter ionizes a small amount of argon in the torch tube; the conductive particles in the torch tube oscillate with the frequency of the magnetic field under the action of the magnetic field, which forms a circular current coaxial with the rectangle tube; atoms, ions, and electrons collide with each other in a strong oscillating motion to produce more electrons and ions; finally, a stable and continuous argon plasma is formed at the torch tube mouth, which fully ionize the sample aerosol that enters the plasma torch flame through the torch tube in a high temperature and inert atmosphere. This ionization source is mainly used for elemental analysis.

4.3.8 Surface ionization (STI)

Surface ionization is also called thermal ionization. Its ionization box is usually equipped with three or two strips of tungsten (or tantalum) which are insulated from each other. The current passing through each strip can be adjusted separately and can be changed within the temperature range of 1 800 K ~ 2 700 K. Coat the sample on the tungsten strip on the side and power on to heat. The center zone temperature is higher than the side zone temperature. Under the action of thermal energy, a part of the analyzed elements is ionized. This ionization source is mostly used for isotope analysis.

v -- the flight speed of ions, in meters per second (m/s).

Therefore, the m/z value of the ion can be determined by its time of flight.

4.5.4 Ion trap mass analyzer

4.5.4.1 It is divided into 3D ion trap and linear ion trap. The core components of different types of ion traps are the electrode systems which are used to generate the electromagnetic field of trapped ions. The difference lies in the electrode structure and the electric field distribution.

4.5.4.2 The principle of the ion trap mass analyzer: for the ions that are formed in the ion source, through the action of the electromagnetic field in the mass analyzer, its movement is limited to a small prefabricated space. By adjusting the electric field parameters, ions of different mass-to-charge ratios enter the "unstable region" in turn, and then separate from the ion trap from the prefabricated space.

The 3D ion trap is composed of a pair of ring electrodes and two hyperbolic end cap electrodes. Add radio frequency voltage or DC voltage again to the ring electrode; ground the upper and lower end cover electrodes. Gradually increase the maximum value of the radio frequency voltage; the ions with a mass-to-charge ratio from small to large gradually enter the unstable region and are discharged through the small holes in the end cap.

The linear ion trap is composed of two sets of hyperbolic rods and two polar plates at both ends. Among the two groups of rods, apply an alternating voltage to one group and two alternating voltages to the other group. Open a slit on one set of the rods; drive the ions out of the slit by changing the three sets of alternating voltages.

4.5.5 Electrostatic field orbitrap mass analyzer

4.5.5.1 The electrostatic field orbitrap mass analyzer is shaped like a spindle; it consists of a spindle-shaped central inner electrode and two outer spindle half electrodes on the left and right.

4.5.5.2 The principle of the electrostatic field orbitrap mass analyzer: gradually add DC voltage to the center electrode to generate an electrostatic field of a special geometric structure in the mass analyzer. After the ions enter the cavity of the mass analyzer, they are attracted by the central electric field and begin to orbit around the center electrode. The ions of different mass-to-charge ratios have different orbital radii; the ions are subjected to vertical centrifugal force and horizontal thrust at the same time; the electrodes oscillate horizontally and vertically along the center. When reaching resonance, the axial reciprocating speed of ions of different mass-to-charge ratios is different; the relationship

measuring instruments, vacuum valves, pipelines, etc., which are used to obtain the vacuum that is required by the instrument.

5 Preparation of the instrument

5.1 General provisions

Before sample analysis, confirm that the instrument is in proper environmental conditions and has good performance; check and confirm that the ion source, detector, recorder, and computer data system are working properly; and the instrument vacuum and all power supplies have met the specified requirements.

5.2 Environmental conditions of instrument operation

The temperature and humidity shall meet the requirements of the instrument; the temperature shall be 20 °C ~ 30 °C; the relative humidity shall usually be less than 70%. Avoid vibration and direct sunlight. Avoid high-concentration organic solvent vapor or corrosive gas in the working environment. The power supply shall meet the regulations; the voltage and frequency of the power supply shall be stable. Avoid interference of various strong magnetic fields and high-frequency electric fields.

5.3 Performance test of the instrument

5.3.1 General provisions

When it has been proved that the design of the instrument can be used for some kind of analysis, it is necessary to judge whether its current state can complete this kind of analysis. There are two mass spectrometer performance test methods that are widely used. One method is the routine inspection of the instrument to determine whether the performance of the instrument is applicable. Another method is to use the mass spectrometer to analyze the test mixture. Both methods can be used; the combination of the two will be better. The latter method is more practical in judging the performance of the mass spectrometer in analyzing the specified samples.

Generally, the contents of sample, background, accuracy, sensitivity, precision, resolution, interference, etc. shall be considered, which facilitates the adjustment.

5.3.2 Sample

The mass spectrometer and its auxiliary components and applied materials must not react with the sample and significantly change the composition of the sample. If there is fractionation in the sampling system or selective adsorption

For a given substance, the response value of the mass spectrometer will change with the short-term factors and the long-term factors. Short-term factors are statistical phenomena that are manifested when the same sample is tested on the same mass spectrometer multiple times on the same day. Long-term factors are statistical phenomena that are manifested in analysis tests which are performed on a mass spectrometer at longer intervals. The evaluation of the analysis method shall include the data of several samples, and consider the possible influencing factors, such as the operator, the time period, the instrument, and the laboratory. This will provide more reliable precision than the single sampling of variable factors.

The precision of the instrument can be determined by repeated scanning of the test mixture.

5.3.7 Resolution

The required resolution of the mass spectrometer depends on the precision, accuracy, and sensitivity that are required by the analytical method, and on the mass number of the measured ions. The resolution capacity of the instrument shall be determined according to the mass range and mass accuracy that are required by the analysis task.

5.3.8 Interference

Sometimes, a mass spectrometer that can provide accurate data can produce inaccurate results, especially when the sample type changes. When one component in the sample mixture influences the mass spectrometer to accurately determine the concentration of other components, it causes interference. This interference is caused by the memory effect of the sample. Different compounds produce different degrees of interference. Usually, harmless compounds also occasionally exhibit interference. If it is believed that there is any interference, clean, bake, leak-check or take other corrective measures for the instrument.

To evaluate the influence of interference effects on accuracy, the most effective test method is the analysis of the test mixture. If an accurate analysis result is obtained, it can be assumed that the condition of the instrument does not interfere with the used test mixture. But for other types of samples, there may still be interference. If the analysis result of the test mixture is not satisfactory, it shall be corrected according to the method that is specified by the instrument. Repeat the analysis to assess the influence of interference until the analysis results are available.

Note: The analysis of the test mixture is the most authoritative indicator of the applicability of the instrument. The preparation of complex, multi-component test mixtures is very difficult, or even impossible; it is also

Possible structure judgment: according to the important low-mass fragment ions, important characteristic ions, molecular ions that are obtained by the mass spectrometer, and the relationship between the parent ion and the daughter fragment ions that are obtained by MS/MS method, linked scanning method, metastable analysis method, perform speculation on possible structures.

According to the inferred molecular structure, compare the standard spectrum with the standard spectrum of similar compounds; or according to the destructive mechanism of the deduced structure compound, make further confirm.

6.3 Result report

If direct sampling is used, a mass spectrum shall be provided.

If chromatography-mass spectrometry is used for sampling, a total ion flow diagram and a mass spectrum for determining the retention time shall be provided.

7 Quantitative analysis

7.1 Sample determination

7.1.1 General provisions

According to the same test conditions as the standard sample, determine the instrument calibration sample, the to-be-tested unknown sample and the background mass spectrum. When the sensitivity of the general method cannot meet the requirements, the multi-ion detection quantitative method can be used.

7.1.2 Quantitative analysis of inorganic elements

Quantitative analysis of inorganic elements that uses mass spectrometry generally requires digestion of the sample before analysis and testing. Digestion methods generally include open container digestion method, closed container digestion method and microwave digestion method. The commonly used reagents for digestion are generally acids, including nitric acid, hydrochloric acid, perchloric acid, sulfuric acid, hydrofluoric acid, and mixed acid of a certain proportion (such as nitric acid: hydrochloric acid = 4:1), and a small amount of hydrogen peroxide. Among them, the interference that is caused by nitric acid is the smallest; it is the first acid for sample digestion. The purity of the reagents shall be above the guaranteed reagent. The used water shall be deionized water (resistivity shall not be less than 18 MΩ·cm).

Common interferences in quantitative analysis can be roughly divided into two categories: one is mass spectrometry type interference, which mainly includes

By preparing no less than 5 standard solutions of non-blank analyte gradient concentration, perform the determination according to the optimized method; record the chromatogram; calculate the peak area of the analyte chromatogram peak. Use the amount of the analyte as the abscissa and the chromatographic peak area of the analyte as the ordinate, to draw a calibration curve. Inject the analysis sample solution under the same conditions; record the chromatogram; calculate the analyte chromatographic peak area; calculate the amount of analyte and the concentration of analyte in the sample from the calibration curve according to the peak area. This method is also called the external standard method; the external standard single-point method is a special case of this method when the horizontal coordinate intercept of the calibration curve is small.

7.2.3 Internal standard method

By preparing a standard solution of analyte gradient concentration of a certain concentration of internal standard, perform the determination according to the optimized method; record the chromatogram; calculate the internal standard and analyte chromatographic peak area. In addition to the blank points, the gradient concentration shall be no less than 5. Use the ratio of the concentration of the analyte and the amount of the internal standard as the abscissa, and the ratio of the analyte and the internal standard chromatographic peak area as the ordinate, to draw the calibration curve. Under the same conditions, inject and analyze the sample solution that is added with the internal standard; record the chromatogram; calculate the internal standard and the analyte chromatographic peak area; use the chromatographic peak area ratio of the analyte and the internal standard to calculate the ratio of the amount of the analyte and the internal standard from the calibration curve; then, from the amount of the added internal standard, calculate the amount of the analyte and the concentration of the analyte in the sample.

7.2.4 Standard addition method

Use the sample solution to add different amounts of analyte standard solutions to prepare no less than 5 sample solutions of gradient concentrations. Perform the determination according to the optimized method; record the chromatogram; calculate the analyte chromatographic peak area. Use the amount of added analyte as the abscissa and the chromatographic peak area of the analyte as the ordinate, to draw a calibration curve; from the intercept of the calibration curve and the abscissa, the analyte concentration in the sample can be calculated.

7.3 Result report

The total ion current diagram, the mass spectrum to determine the retention time, as well as the calibration curve and correlation coefficient shall be

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